Universidade de Lisboa

Faculdade de Medicina Dentária

Reparability of two different CAD/CAM polymer materials using a light-cured composite and Universal adhesives.

Bianca Andrada Rosca

Dissertação

Mestrado Integrado em Medicina Dentária

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Dissertação orientada pelos

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Mestrado em Medicina Dentária

2016

To my parents,

The most important thing in my life…

Table of contents

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Abstract

Objective: The aim of the present study was to evaluate the reparability of two different CAD/CAM polymer materials, polymethyl methacrylate (PMMA) and polyoxymethylene (POM) by assessing the shear bond strength (SBS) of a light-cured composite, using four different adhesive systems.

Materials and Methods: Eighty blocks of PMMA and eighty blocks of POM, with 7 mm of diameter and 4 mm height, were milled by the manufacturer. The top surface of all specimens was abraded with 50 µm Al_2O_3 and after, ultrasonically cleaned in an ethanol ultrasonic bath for 5 minutes. The 80 specimens of each material were randomly allocated to four experimental groups (n=20) according to the different adhesive system used: OptiBond™ XTR (Kerr™, Italia) Futurabond® M+ (VOCO GmbH, Germany), Scotchbond™ Universal (3M ESPE, Germany) or OptiBond™ Solo Plus (Kerr™, Italia). The bonding area was customized using a perforated adhesive tape with round-shaped orifices (3 mm diameter) and each adhesive was applied according to the manufacturer´s recommendations. The repairing composite resin Grandio®SO (VOCO GmbH, Germany), was applied according the manufacturer's instructions, and polymerized during 10 seconds (1000 mW/cm^2). Specimens were stored in distilled water at 37 °C for 48 hours before the SBS was tested and failure modes were assessed.

Results: The mean SBS yielded with PMMA specimens was significantly higher than with POM. There were no statistical differences between adhesive systems. No cohesive failure was obtained, with the POM specimens having mostly adhesive failures, in contrast with the PMMA with mostly mixed failures and no significant difference was identified between adhesives.

Conclusion: It is possible to achieve an adequate adhesion bond to POM and PMMA. SBS values were higher in PMMA specimens than to POM but no differences were found between adhesive systems.

Keywords: PMMA; POM; Acetal Resin; CAD/CAM polymer; Reparability; Universal Adhesive.

Resumo

Introdução: Polimetilmetacrilato (PMMA) e polioximetileno (POM) são polímeros com propriedades físicas e mecânicas otimizadas além de apresentarem elevada biocompatibilidade. A sua polimerização em condições laboratoriais padronizadas e posterior fresagem através do sistema CAD/CAM permite o fabrico de restaurações protéticas provisórias, pilares estéticos de implantes e ganchos estéticos de próteses removíveis. O facto de poderem ser utilizados para a confeção de restaurações fixas provisórias de longa duração, leva a que muitas vezes seja necessário a adição de outro material. Tal procedimento poderá ser importante, tanto para reparação em caso de fratura ou desgaste da restauração, ou quando se pretende alterar a sua forma como forma de cumprir os objetivos da reabilitação. Para a reparação ou alteração deste tipo de restaurações provisórias, a resina composta é normalmente o material elegido pelos clínicos, especialmente devido à sua fácil utilização e propriedades estéticas. Existe escassa informação sobre qual o melhor procedimento para obter valores de adesão adequados entre o material a reparar e o material reparador. A obtenção de elevados valores de adesão está normalmente dependente de uma componente mecânica e componente química. entre os dois materiais é normalmente criada por dois mecanismos, um mecânico e outro químico. Se existe algum consenso quanto ao método de condicionamento mecânico da superfície a reparar, aplicação de jato de óxido de alumínio, não existem estudos conclusivos sobre o tipo de sistemas adesivos a utilizar. Recentemente, foi disponibilizado um novo grupo de sistemas adesivos, os adesivos universais, cujos fabricantes reclamam eficácia no estabelecimento de adesão a diversos substratos. No entanto, tal eficácia em promover adesão aos polímeros utilizados com o sistema CAD/CAM não se escontra estudado.

Objetivos: O objetivo do presente estudo foi avaliar a reparabilidade de dois materiais poliméricos utilizados para fresagem através do sistema CAD/CAM, o polimetilmetacrilato (PMMA) e o polioximetileno (POM), utilizando um compósito fotopolimerizável e diferentes sistemas adesivos, de acordo com as seguintes hipóteses nulas: 1) o tipo de material polimérico utilizado como substrato não influencia a resistência adesiva do material reparado; 2) não existe diferença entre a resistência adesiva promovida pelos diferentes sistemas adesivas utilizados.

Materiais e Métodos: Um total de 160 blocos, 80 blocos de PMMA e 80 blocos de POM, foram fresados pelo fabricante, com dimensões padronizadas de 7 mm de diâmetro e 4 mm de altura. O topo de cada um dos espécimes foi condicionado mecanicamente com jato de Al_2O_3 (granulometria - 50 µm; pressão - 2,5 bar; distância - 10 mm; tempo - 15 segundos). Após o condicionamento mecânico, todos os espécimes foram lavados num banho ultrassónico de etanol durante 5 minutos. A superfície dos espécimes foi então lavada com jato de água e ar, durante 15 segundos e, por fim, seca com jato de ar durante 10 segundos. Os 80 espécimes de cada material foram então aleatoriamente atribuídos a um dos quatro grupos experimentais (n=20), segundo o sistema adesivo utilizado: OptiBond™ XTR (Kerr™, Itália) Futurabond® M+ (VOCO GmbH, Alemanha), Scotchbond™ Universal (3M ESPE, Alemanha) ou OptiBond™ Solo Plus (Kerr™, Itália). Os espécimes foram fixados a placas de Watanabe com gesso, e a área de adesão (3 mm de diâmetro) foi estabelecida utilizando uma fita adesiva perfurada. O sistema adesivo foi em seguida aplicado e fotopolimerizado de acordo com as instruções do respetivo fabricante. Por fim, o compósito Grandio®SO (VOCO GmbH, Germany), foi aplicado num incremento único de 2 mm e polimerizado, consoante as instruções do fabricante, durante 10 segundos, utilizando um fotopolimerizador (Ortholux LED Curing Light - 3M Unitek, Alemanha) com uma intensidade de 1000 mW/cm^2. A própria placa de Watanabe foi utilizada como molde para a aplicação do compósito e a ponta do fotopolimerizador foi sempre aplicada de forma perpendicular à superfície de união de maneira a garantir o máximo possível de energia. Após o procedimento adesivo, todos os espécimes foram armazenados em água destilada, a 37 º C durante 48 horas. Findo este período de tempo de armazenamento, foram realizados os testes mecânicos. A resistência adesiva a tensões de corte foi medida com uma máquina de testes universal (Instrom modelo 4502, Instrom Ltd., Bucks, HP 12 3SY, England), utilizando uma célula de carga de 1 kN e uma velocidade de 0,5 mm/min. Após o ensaio de resistência mecânica, a superfície de fratura foi observada com um estereomicroscópio (EMZ-8TR, Meiji Techno Co) utilizando uma ampliação de 20x com o objetivo de avaliar o tipo de falha de união. O tipo de falha foi classificado em três tipos possíveis. Falha adesiva, quando a falha ocorreu na interface adesiva. Falha coesiva, caso a falha tenha ocorrido dentro do compósito ou do substrato. Falha mista, caso se tenha verificado uma combinação das duas situações anteriormente descritas. Os dados foram submetidos a análise estatística de acordo a natureza da variável independente em questão. Após a verificação da normalidade da distribuição da amostra e da homogeneidade da variância, realizada recorrendo

aos testes de Shapiro-Wilk ($p > 0,05$) e Levene ($p > 0,05$), respetivamente, os dados de resistência adesiva a tensões de corte foram submetidos a uma análise de variância (ANOVA de duas vias) utilizando o tipo de substrato e o tipo de adesivo utilizado como fatores independentes. Devido à natureza dos dados, foram utilizados testes não paramétricos, segundos métodos de Kruskal-Wallis e Mann-Whitney, para a análise do tipo de falha. Para todos os testes, foi fixada uma significância estatística de 5% (alfa = 0,05).

Resultados: Os valores médios de resistência adesiva a tensões de corte variaram entre 22,6 MPa e 40,0 MPa. Os valores médios mais baixos foram obtidos quando o substrato a reparar foi o POM e o adesivo utilizado foi o OptiBond® Solo Plus™. Os valores de resistência adesiva mais elevados foram obtidos no grupo de espécimes fabricados com o PMMA e reparados utilizando o adesivo OptiBond™ XTR. A análise estatística dos dados obtidos permitiu verificar que o tipo de material polimérico utilizado como substrato influenciou de uma forma estatisticamente significativa ($p < 0,001$) os valores de adesão. A resistência adesiva obtida nos espécimes fabricados com o PMMA apresentou valores mais elevados que os obtidos nos espécimes fabricados com o POM. No entanto, não se observaram diferenças estatisticamente significativas ($p = 0,062$) entre valores de resistência adesiva obtidos com os diferentes sistemas adesivos utilizados. A interação entre os dois fatores (substrato e adesivo) também não se revelou com significado estatístico ($p = 0,212$). Quanto ao tipo de falha de união observado, a maioria das falhas obtidas com o PMMA foram do tipo misto (86,3%). Ao contrário, com o POM o tipo de falha de união mais observado foi o adesivo (87,5%). A análise estatística inferencial, permitiu detetar diferenças estatisticamente significativas ($p < 0,001$) entre o tipo de falha observado em cada um dos dois materiais poliméricos de CAD/CAM. No entanto, tal como tinha sucedido para a resistência adesiva, também não se verificaram diferenças estatisticamente significativas ($p=0,925$) em relação ao tipo de falha observado com os diferentes sistemas adesivos utilizados.

Conclusões: O presente ensaio laboratorial permitiu verificar que é possível obter uma adesão eficaz a qualquer um dos materiais poliméricos de CAD/CAM testados. No entanto, os valores de adesão foram mais elevados para o PMMA do que para o POM, independentemente do sistema adesivo utilizado. Por outro lado, nas condições experimentais do presente estudo, os sistemas adesivos universais testados não se mostraram mais eficazes que o sistema adesivo convencional utilizado como controlo.

Palavras-chave: PMMA; POM; Resina acetálica; Polímeros de CAD/CAM; Reparabilidade; Adesão.

Introduction

Fixed temporary or provisional dental restorations are used for a limited period of time, after which, they are replaced by the definitive ones (The Academy of Prosthodontics, 2005). They are meant to enhance aesthetic, and/or function, protect pulpar and periodontal health, promote guided tissue healing, prevent migration of the adjacent teeth and provide an adequate occlusal scheme (Verri *et al*., 2008; Peñate *et al*., 2015; Proussaefs *et al*.,2015). They are also diagnostic tools, as they permit the evaluation of hygiene procedures and maxillo-mandibular relationships, playing an important role for understanding the treatment outcomes and limitations. (Amet *et al*., 1995; Lodding, 1997; Gratton *et al*., 2004; Reshad *et al*., 2010; Kurbad, 2014)

Several types of self or light curing acrylic resins, such as polymethyl methacrylate (PMMA) resin, polyethyl methacrylate resin, polyvinyl methacrylate resin, bis-acryl composite resin, and visible light-cured urethane dimethacrylates, are the most common materials used for the fabrication of interim restorations (Michalakis *et al*, 2006; Patras *et al*, 2012; Prasad *et al*., 2014).

Despite provisional restorations mainly serving for a short period of time, some situations such as comprehensive occlusal reconstructions or oral implantation treatments require a long-term temporary restoration (Beuer *et al*., 2011; Güth *et al*., 2012; Bähr *et al*, 2013). Therefore, the material used to fabricate these type of restorations should present improved mechanical properties, adequate colour stability and provide marginal integrity to face extended functional loading. (Ergün *et al*., 2005; Rayyan *et al*, 2015;)

For this purpose, the use of resin materials for computer aided design/computer aided manufacturing (CAD/CAM) have increased lately (Vanoorbeek *et al*., 2010; Edelhoff *et al*., 2012). Nowadays, temporary and even permanent dental restorations can be milled of polymeric materials, such as polymethyl methacrylate (PMMA)-based or polyoxymethylene (POM), CAD/CAM blocks. (Wimmer *et al*., 2016).

Several studies have shown that the milling technique provide high precision restorations, with a more precise marginal quality than conventionally processed resin, improved colour stability and better physical and mechanical properties compared to the conventionally fabricated ones. (Alt *et al*., 2011; Khng, 2013; Rayyan *et al*, 2015; Peñate *et al*. 2015)

Restorations milled of PMMA-based CAD/CAM blocks present better mechanical properties, a wider range of translucency and a higher stability against discolouration compared to the conventionally polymerized ones. (Wiegand *et al.*, 2015)

Polyoxymethylene (POM), also called "Acetal Resin", is an alternative to PMMA-based resin. It is a thermoplastic polymer with a monomer-free crystalline structure consisting of a chain of alternating methyl groups linked by an oxygen molecule, and it is produced by the polymerization of formaldehyde (Fitton *et al*., 1994; Turner *et al*., 1999; Tannous *et al*., 2012; Maha *et al.*, 2015).

This material has been brought into medicine in total hip replacement surgeries and temporomandibular joint reconstructions, with its use in dentistry increasing lately due to its mechanical and physical properties such as high wear, fracture and creep resistance and high fatigue endurance and its superior esthetic features (Thomas *et al.*, 2011; Cervera der Rio, 2013; Tannous *et al*., 2012; Maha *et al.*, 2015). Being monomer-free allows it to be used as an innovative and a safe treatment alternative for patients who are allergic to conventional resins, presenting high biocompatibility (Lekha *et al*, 2016). In addition, is a hydrophobic material, which means that does not absorb water or saliva and has little or no porosity, which reduces the accumulation of biological material like plaque, avoiding odour and stains (Maha *et al*., 2015).

At the moment is used as a material for pre-formed clasps for partial dentures, as direct retainers attached to a cobalt-chromium removable partial denture framework, removable partial prostheses, partial denture frameworks, provisional bridges and restorations, post-surgery space maintainers, occlusal splints, and even implant abutments. (Thomas *et al*, 2011; Tannous *et al*., 2012; Cervera der Rio, 2013; Maha *et al*, 2015; Lekha *et al*, 2016)

As they can be used for a long period of time, repairing or relining temporary restorations is common in clinical practice, since fractures may occur intraorally between appointments. (Chen *et al*., 2008; Patras *et al.*, 2012; Wiegand *et al.*, 2015). Furthermore, those materials should undergo reshaping, addition, removing and repolishing procedures to allow new treatment approaches (Güth *et al*., 2012; Patras *et al.*, 2012; Bähr *et al.*, 2013).

Light-cured resin materials have been suggested to repair and reshape provisional restorations, because of their adequate working time, easy procedure and aesthetic advantages

(Patras *et al.*, 2012). However, the strength of this bonding remains questionable and scarce information on the reparability of CAD/CAM polymer-based materials and the preferred repair method is available so far (Wiegand *et al*., 2015).

Previous studies showed that mechanical pre-treatment such as surface roughening through air abrasion with aluminium oxide before the bonding procedure increases the bond strength repair of all the materials. (Wiegand *et al*., 2015) Actually, the limiting factor is the low chemical bond strength to the polymeric material since the standardized polymerization under high pressure and high temperature results in a higher degree of conversion and less residual monomer in the material. (Bähr *et al*., 2013). It is necessary to assess if the use of Universal adhesive might be a solution to overcome the difficulty in achieving adequate chemical bonding, since it is known their ability to bond various materials is well known (Alex, 2015).

Objectives

Therefore, the aim of the present study was to evaluate the reparability of two different CAD/CAM polymer materials (PMMA and POM) by assessing the shear bond strength (SBS) of a light-cured composite, using four different adhesive systems, according to the following null hypothesis:

1) there are no differences between the bond strength to the CAD/CAM polymer materials;

2) the SBS is not influenced by the adhesive system used.

Materials and Methods

Based on a pilot study, the sample size (n=20) was estimated with a power analysis in order to provide statistical significance (α=0.05) at 80% power.

One hundred and sixty blocks of CAD-CAM polymeric materials, with standardized dimensions of 7 mm diameter and 4 mm height, were milled by the manufacturer. Eighty blocks were made of PMMA (PMMA – GT Medical S.L, Spain) and 80 were made of POM (POM-fit – GT Medical S.L, Spain) (Figure 1) (Table 1).

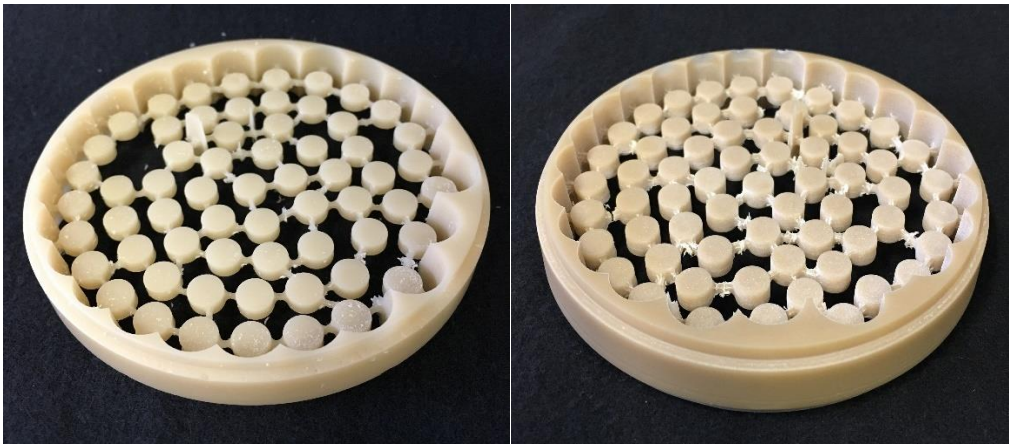

Figure 1: CAD/CAM milled blocks made of PMMA (left) and POM (right).

Material	Manufacturer	Composition*
PMMA	GT Medical, Madrid, Spain	Powder (Polymethylmethacrylate); Liquid (Methyl Methacrylate MMA and Ethyleneglycol Dimethacrylate EDMA); Pigments (yellow – FeOCH, Black – Fe3O4, White – TiO3, Red – Fe2O3).
POM-FIT	GT Medical, Madrid, Spain	Acetal resin (Polyoxymethylene)

**According to the information provided by the manufacturer.*

Table 1: Characteristics of the polymeric CAD/CAM materials used.

The top surface of all specimens was mechanically conditioned with 50 μm Al_2O_3 (Microetcher II; Danville Engineering) airborne particle abrasion, performed perpendicularly to the block surface with 2.5 bar pressure for 15 seconds at 10 mm distance (Figure 2). After the sandblasting, the specimens were ultrasonically cleaned in an ethanol ultrasonic bath (Elmasonic One; Elma) for 5 minutes and then cleaned with water-spray and air-dried carefully with oil-free compressed air.

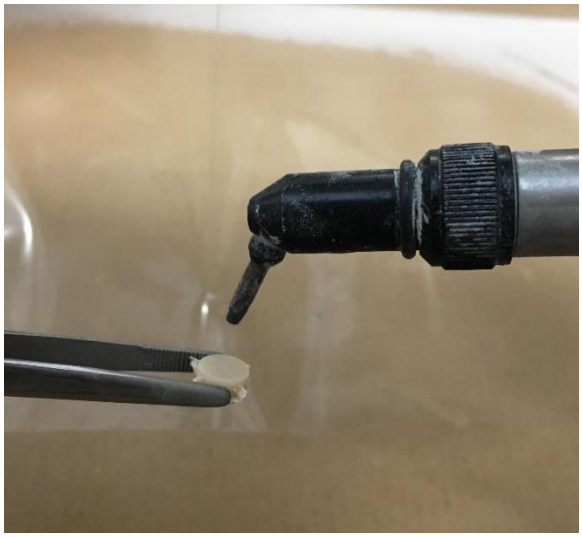

Figure 2: Sandblasting of the polymer blocks.

The 80 specimens of each material were randomly allocated to four experimental groups, 20 specimens each, according to the different adhesive system used (Figure 3): OptiBond™ XTR (Kerr™, Scafati, SA 1-84018, Italia) Futurabond® M+ (VOCO GmbH, Cuxhaven, 27472, Germany), Scotchbond™ Universal (3M ESPE, Neuss, 41453, Germany) or OptiBond™ Solo Plus (Kerr™, Scafati, SA 1-84018, Italia) (Table 2).

The specimens were fixed on Watanabe plates using plaster and to customize and define the bonding area, a perforated adhesive tape (Adhesive vinyl, SRA3; Xerox) with round-shaped orifices (3 mm in diameter) was positioned on the surface of the blocks (Figure 4). Each adhesive was applied according to the manufacturer´s recommendations (Table 2).

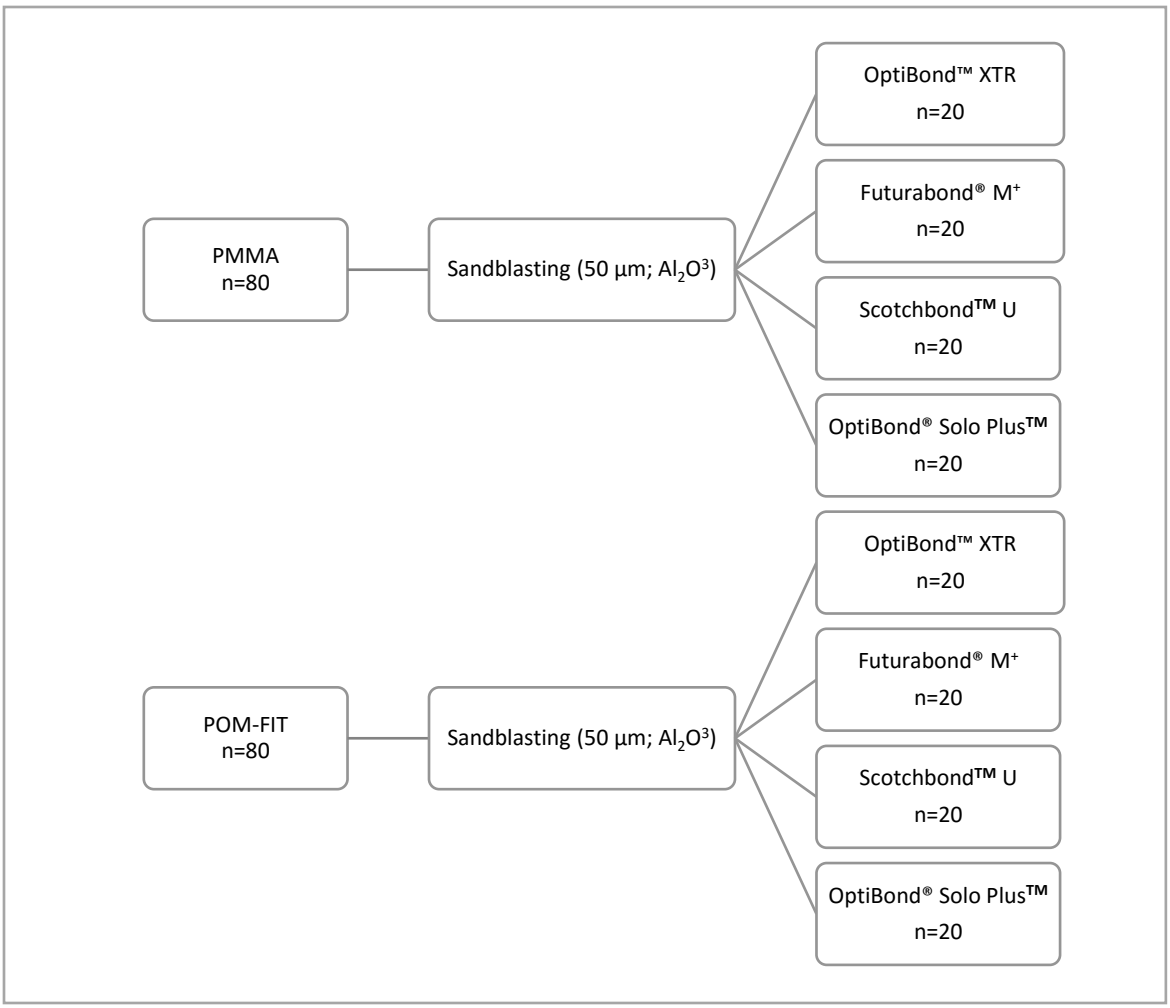

Figure 3. Experimental design protocol.

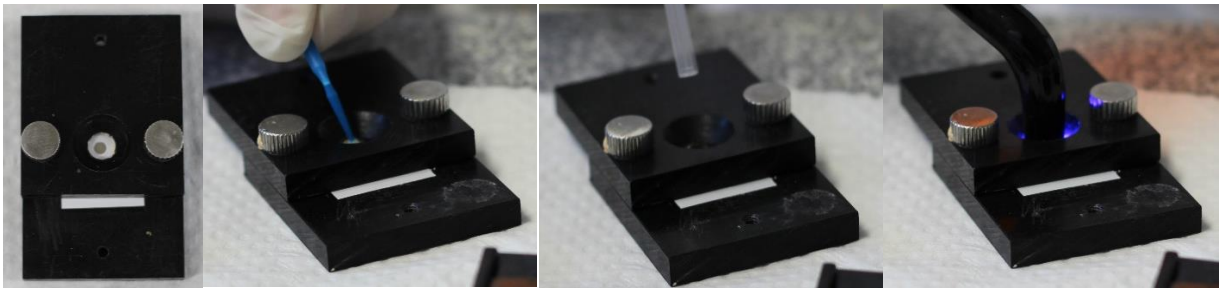

Figure 4. Customization of the adhesive area and adhesive procedure.

Adhesive	Batch Nr	Manufacturer	Composition*	Bonding Procedure**
OptiBond® XTR™	Adhesive: 5812210 Primmer: 5790784	Kerr™, Orange, CA, USA	Primer – GPDM phosphate monomer, acetone, ethyl alcohol, HEMA Adhesive – ethyl alcohol, alkyl dimethacrylate resins, barium aluminoborosilicate glass, fumed silica, sodium hexafluorosilicate	Apply primmer using scrubbing motion (20 sec). Air thin with medium air pressure (5 sec). Apply the adhesive with brushing motion (15 sec). Air thin with medium air pressure and then strong air (5 sec). Light-cure during 10 sec.
Futurabond® M$^+$	1612531	VOCO GmbH, Cuxhafen, Germany	Bis-GMA, 2-hydroxyethyl methacrylate, HDDMA, Acidic Adhesive Monomer, Urethanedimethacrylate, pyrogenic silicic acids, catalyst.	Apply the adhesive with brushing motion (20 sec). Air thin with medium air pressure and then strong air (5 sec). Light-cure during 10 sec.
Scotchbond™ Universal	616836	3M ESPE, St. Paul, MN, USA	MDP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond™ Copolymer, Filler, Ethanol, Initiators, Water, Silane.	Apply the adhesive with brushing motion (20 sec). Air thin with medium air pressure and then strong air (5 sec). Light-cure during 10 sec.
OptiBond® Solo Plus™	5357097	Kerr Corporation, Orange, CA, USA	Bis-GMA, GDM, HEMA, GPDM, Ethanol, Aluminiun borosilicate glass, Fumed silica, Sodium hexafluorosilicate, Photoinitiator	Apply the adhesive with brushing motion (20 sec). Air thin with medium air pressure and then strong air (5 sec). Light-cure during 20 sec.

*Table 2: Characteristics of the adhesive systems and the bonding procedure. *According to the information provided by the manufacturer. **According manufacturer's instructions.*

The repairing composite resin Grandio®SO (VOCO GmbH, Cuxhaven, 27472, Germany), was applied according the manufacturer's specific instructions, in a 2 mm layer, using the Watanabe plate as a mold, and polymerized during 10 seconds.

The adhesive and composite were light cured using Ortholux LED Curing Light (3M Unitek, 82171 Puchheim, Germany, Lot nr: 939830000776). The tip of the light curing unit was kept perpendicular to and in contact with the surface in order to receive a maximum curing energy. The intensity of the curing light, 1000 mW/cm^2, was monitored with a Cure Rite Visible Curing Light Meter (Dentsply, York, PA, USA).

Specimens were stored in distilled water at 37 °C for 48 hours before testing. SBS was measured with a single-plane lap device in a universal testing machine Instron model 4502 (Instrom Ltd., Bucks, HP 12 3SY, England) with a 1 kN load cell at a cross-head speed of 0.5 mm/min (Figure 5).

The mode of failure was analyzed with a stereomicroscope (EMZ-8TR, Meiji Techno Co) at 20x magnification (Figure 6). The failure mode was classified as adhesive type, if the failure occurred at the adhesive interface, cohesive when the failure occurred within the composite or CAD/CAM resin, or as mixed if a combination of failures occurred.

Figure 5: Measuring the SBS: Instrom Testing machine.

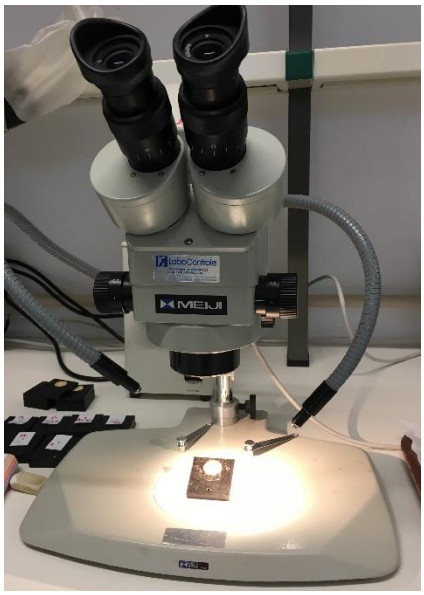

Figure 6. Stereomicroscope EMZ-8TR.

Data were statistically analyzed with software (IBM SPSS Statistics 20; SPSS Inc). After assessing normality and homoscedasticity with Shapiro–Wilk and Levene's tests ($p>0.05$), SBS data were submitted to 2-way ANOVA model ($\alpha=0.05$). Non-parametric Kruskal-Wallis and Mann-Whitney tests were used to analyze the failure mode data ($\alpha=0.05$).

Results

The descriptive statistic of the sample is presented in Table 3, with the SBS values (mean and standard deviation) and Failure Mode (number and percentage) according to each experimental group.

The mean SBS ranged between 22.6 MPa for the POM specimens repaired with OptiBond Solo Plus and 40.0 MPa for the PMMA with OptiBond XTR.

The mean SBS yielded with PMMA specimens was significantly ($p<0.001$) higher than with POM (Figure 7). Two-way ANOVA showed no statistical ($p=0.062$) differences between adhesive systems (Figure 8) and an interaction ($p=0.212$) between the two factors was not detected (Table 4).

When the failure mode was analyzed, no cohesive failure was obtained, regardless of the experimental group considered. A statistically significant difference was detected between the failure mode observed with the two CAD/CAM resins ($p<0.001$), with the POM specimens having mostly adhesive failures (87.5%), in contrast with the PMMA with mostly mixed failures (86.3%) (Figure 9). No statistically significant ($p=0.925$) difference was identified between adhesives (Figure 10).

Polymeric material	Adhesive	SBS values (MPa)		Failure Mode N (%)		
		Mean	SD	Adhesive	Mixed	Cohesive
POM	OptiBond™ XTR	24,3	3,56	18 (90%)	2 (10%)	0 (0%)
	Futurabond® M+	26,2	4,23	18 (90%)	2 (10%)	0 (0%)
	Scotchbond™ Universal	23,3	3,93	18 (90%)	2 (10%)	0 (0%)
	OptiBond™ Solo Plus	22,6	2,82	16 (80%)	4 (20%)	0 (0%)
PMMA	OptiBond™ XTR	40,0	3,47	1 (5%)	19 (95%)	0 (0%)
	Futurabond® M+	30,3	4,66	2 (10%)	18 (90%)	0 (0%)
	Scotchbond™ Universal	30,7	3,83	2 (10%)	18 (90%)	0 (0%)
	OptiBond™ Solo Plus	29,4	3,58	6 (30%)	14 (70%)	0 (0%)

Table 3: Descriptive statistic of the SBS values and Failure Mode distribution according to the 8 experimental groups.

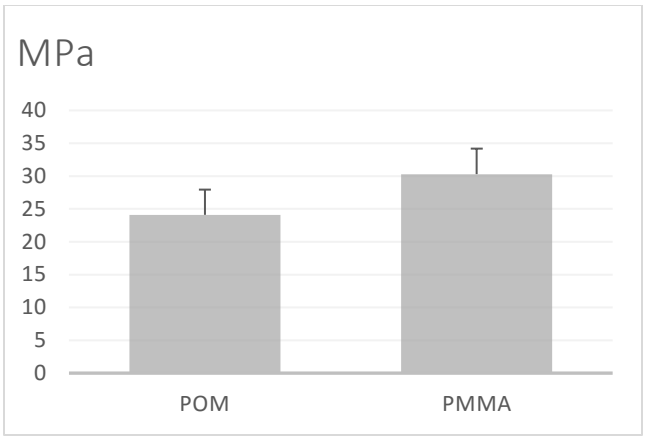

Figure 7. Mean SBS values and standard. deviation of the CAD-CAM polymeric material. Statistically significant difference was found between materials (p<0.,001).

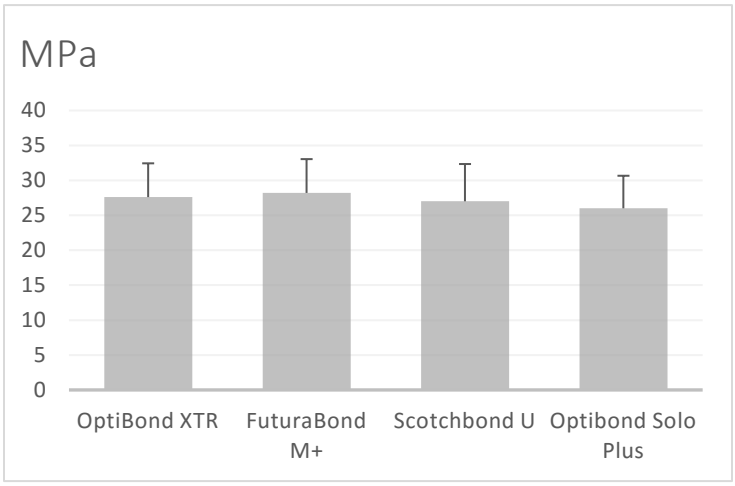

Figure 8. Mean SBS values and standard. deviation according to the adhesive protocol system used. No statistically significant difference was found (p>=0.,0625).

Source	df	Sum of squares	Mean Square	F	Sig.*
Material	1	1535.059	1535.059	106.670	0.000
Adhesive	3	107.597	35.866	2.492	0.062
Material/ Adhesive	3	65.584	21.861	1.519	0.212
Error	152	2187.400	14.391		
Total	160	122350.021			

*Table 4. Two-Way ANOVA. *p<0.05 indicates statistically significant differences.*

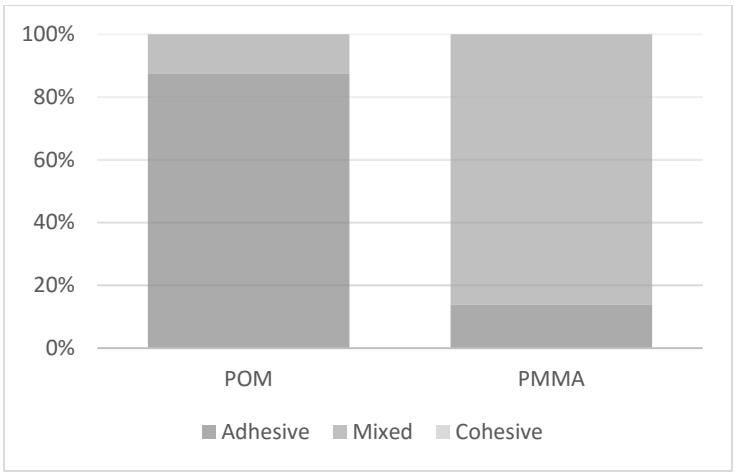

Figure 9. Failure Mode distribution according to the CAD-CAM polymeric material. A statistically significant difference was shown between the groups ($p<0.001$).

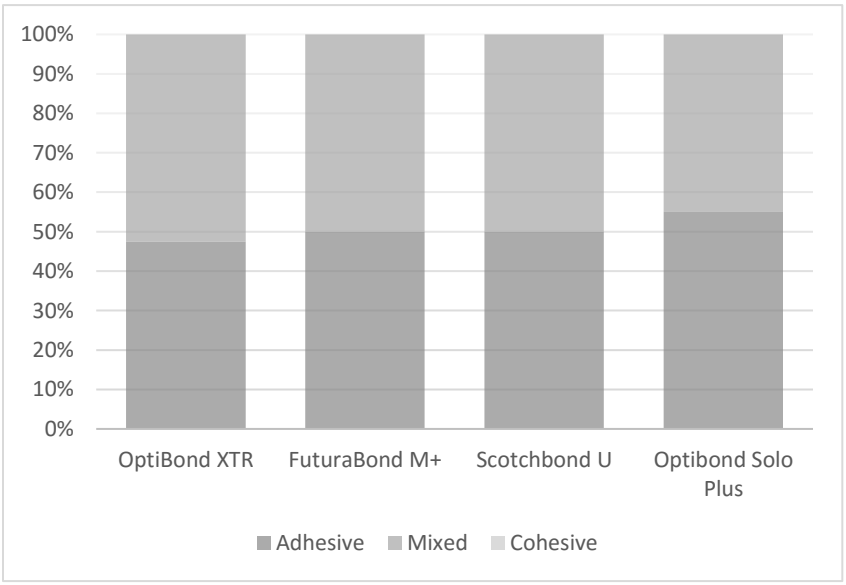

Figure 10. Failure Mode distribution according to the adhesive system used. No statistically significant difference was identified between the adhesives ($p=0.925$).

Discussion

The SBS test has been widely used (Heintze, 2013), although some authors suggest there are problems related to the validity of the obtained measurements, sustaining that the stresses are mostly concentrated in the substrate and not in the adhesive interface, thus causing its premature failure prior to the interface itself, resulting in a higher percentage of cohesive failures (Della Bona *et al*., 1995; Meerbeek *et al*., 2010). However, even with the high SBS values obtained in the present study, when the failure mode was analyzed, no cohesive failure was obtained, regardless the experimental group considered, which might confirm the validity of the measurements. Moreover, this method has been used in most of the recent studies testing the adhesion to CAD/CAM materials, enabling the comparison of the present results (Wiegard *et al*., 2015; Keul *et al*., 2014; Bähr *et al*., 2013)

With the advent of adhesive dentistry, it has become possible to bond artificial materials to other artificial materials which enables dentists to repair rather than replace restorations in case of chippings, fractures or marginal caries (Chen *et al*., 2008; Heintze *et al*., 2013). Another approach made possible by adhesion is the reshaping of the restorations by adding another material, in case of aesthetic improvements required by the patient (Güth et al., 2012; Patras *et al*., 2012; Chen *et al*., 2008).

For the CAD/CAM materials considered in this study, no information on the preferred repair method or material is available so far, especially regarding the acetal resin. (Patras *et al*., 2012; Stawarczyk *et al*., 2013; Wiegand *et al*., 2015) The chemical composition of the base and the added material, surface characteristics of the interface, and time elapsed since the initial setiing of the restoration should be considered before choosing the repair procedure (Patras *et al*., 2012)

Self-cured acrylic resins made from PMMA, bis-acryl provisional resins and resin composites, are the most common materials used for repair, because they have the ability to easily reconstruct shape defects, allowing for simple and quick manipulation (Chen *et al*., 2008). Among those, the use of self-cured acrylic resins is most popular, although direct clinical repair procedures with PMMA can be technically challenging due to the unpleasant odour, significant shrinkage, short working times and a pronounced exothermic setting reaction (Patras *et al*., 2012). Moreover, residual methacrylate monomer used as repair or relining materials demonstrates cytotoxicity and

potential allergenicity (Chen *et al*., 2008; Patras *et al*., 2012). More recently, light-cured resin composites have been suggested for the intraoral repair of provisional restorations with the following advantages: availability in numerous shades and viscosities, ease of application and manipulation, adequate working time, minimal odour, low polymerization shrinkage, and increased marginal accuracy (Chen *et al*., 2008; Patras *et al*., 2012; Blum *et al*., 2014).

Since several studies showed that previous mechanical conditioning of the substrate is important to achieve high bond strength (Patras *et al*., 2012; Stawarczyk *et al*., 2012; Stawarczyk *et al*., 2013; Keul *et al*., 2014; Wiegand *et al*., 2015), the specimens were all sandblasted with aluminium particles (50μm) before the bonding procedure in the present investigation.

Nevertheless, the ability of achieving an adequate chemical bonding to the substrate remains questionable, since the polymerization under standardized conditions, high pressure and high temperature, of the CAD/CAM materials, results in a higher degree of conversion and less residual monomer in the material (Bähr *et al*., 2013). Thus, the amount of residual monomer or free radicals is very low or even insufficient to allow co-polymerization and these materials present a low surface energy (and wettability) and resistance to surface modification by different chemical treatments (Li Zhoua *et al.*, 2014). Moreover, monomers of the adhesive system (Bis-GMA, HEMA; GDMA) and of the composite (Bis-GMA, UDMA, TEGDMA, bis-EMA) might not co-polymerize with PMMA-based materials (Stawarczyk *et al*., 2012; Wiegand *et al*., 2015).

Several possible mechanisms were proposed to explain the role played by the adhesive during the composite resin filling repair. Firstly, the micromechanical interlocking formed by infiltration of the monomer into micro retentions created by the mechanical conditioning should be considered, since the adhesive monomers enable the achievement of better wetting of the surface, due to the solvent and a surfactant that are often added to the bonding agent and the wetting properties of the adhesive monomers themselves, since they have low viscosity (Marshall *et al*., 2010; Seung-Ryong *et al*., 2016). The POM specimens presented mostly adhesive failures, in contrast with the PMMA with mostly mixed failures, indicating that the bond strength achieved is higher to the PMMA. Thus the first null hypothesis that there were no differences between the two substrates was rejected. Since the micromechanical interlocking plays an important role in the repair bond strength, the differences obtained might be explained by differences in the surface

properties of the two substrates: it is known than the POM presents a higher resistance to surface modification (Maha *et al*., 2015).

In addition to the mechanical interlocking, it was suggested the possibility of a chemical bond formation to the surface fillers and to the matrix (Bähr *et al*., 2013). Although, as mentioned before, a co-polymerization with the traditional adhesive monomers is not sufficient because the CAD/CAM resins are industrially polymerized and present a higher degree of conversion and low amount of unsaturated C-C bonds, it is known that the new Universal adhesive systems contain silane or phosphoric acid monomers with high affinity to inorganic filler particles, that may form covalent bond to the unreacted methacrylate groups on the matrix or to the inorganic compound of the CAD/CAM materials (Cardoso *et al*., 2011; Stawarczyk *et al*., 2015; Alex, 2015; Seung-Ryong *et al*., 2016).

However, in the present study, for the different adhesives, although there are differences in their composition (OptiBond® Solo Plus™ does not contain phosphoric monomers) no statistically significant difference was reached, indicating that the bonding agent doesn't influence the failure mode, which means that the second null hypothesis cannot be rejected. Further studies are necessary, with no mechanical conditioning (highly polished specimens), to assess if the chemical bonding alone is sufficient to achieve adequate SBS values.

Also, all surfaces were air-abraded to create a standardized surface with some micromechanical retentions. Different conditioning procedures, like abrasion with alumina-coated silica particles followed by silanization, should also be investigated, since there are several pre-treatment procedures available and described by the literature as efficient to condition CAD/CAM polymeric material (Schmidlin *et al*., 2010; Hallmann *et al*., 2012; Li Zhoua *et al*., 2014; Wiegand *et al*., 2015; Seung-Ryong *et al*., 2016).

From a methodological point of view, a shortcoming of this study is the lack of artificial ageing by thermocycling or long-term water storage. In previous studies, adhesion between PMMA-based polymer restorations and conventional resin cements, as well as shear bond strength of repaired dimethacrylate-based direct composites, has shown to be impaired by ageing (Ozcan *et al*., 2010; Bähr et al., 2013; Wiegand *et al*., 2015). Several mechanisms were proposed to explain that. Some state that thermocycling ageing leads to mechanical stress on the bonding area of the

repaired substrate and the residual monomer content and consequently the number of carbon-carbon double bonds, but it is also discussed that thermocycling might increase the repair bond strength by intensifying the process of post-polymerization between polymeric CAD/CAM materials and adhesive resins (Bähr *et al*., 2013).

To the knowledge of the author this is the first evaluation of the bonding performance to POM in regard to dental applications. Hence, the present results cannot be compared to other available studies. In addition, differences in the methodology and especially the substrate evaluation, adequate comparisons and conclusions are significantly impeded. However, as this was the first study, the main goal was to assess the overall viability of establishing bonding to POM.

Likewise, very few in vitro studies address the repairation of restorations milled of PMMA-based CAD/CAM blocks and consequently, many of the comparisons and conclusions drawn about this matter are based on essays studying the bonding strength to these kind of polymers regarding different conditioning and adhesive procedures for veneering or cementing purposes.

Techniques and materials used in this study should be tested in more demanding laboratory conditions to closer simulate clinical environment.

Although it is difficult to set a clinically relevant SBS value for bond strength after repair, most in vitro studies presented repair bond strength values of dimethacrylate-based direct composites of at least 20 MPa, depending on the kind of composite material and the repair method used (Wiegand *et al.*, 2015). In the present study, the mean SBS values obtained were higher than those suggested above, demonstrating that the repairing method tested is efficient, with the PMMA blocks achieving statistically significant higher values than the acetal resin, regardless of the adhesive system used.

Conclusions

It is possible to achieve an adequate adhesion bond to POM and PMMA. However, SBS values were higher in PMMA specimens than to POM. No differences were found between adhesive systems.

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Appendices

List of Figures and Tables

Data from experimental group: POM + OptiBond™ XTR

N	Force (N)	MPa	Failure Mode*	Simplified Failure mode**
1	153.1	23.07	1	1
2	153.8	21.76	1	1
3	156.3	22.11	1	1
4	202.1	28.59	1	1
5	199.5	28.22	1	1
6	175.3	24.8	1	1
7	180.2	25.49	1	1
8	155.3	21.97	5	2
9	127.4	18.02	1	1
10	184.3	26.07	1	1
11	198.7	26.98	1	1
12	120.5	17.05	1	1
13	182.2	25.78	1	1
14	160.1	22.65	5	2
15	160.9	22.76	1	1
16	155.4	21.98	1	1
17	158	22.35	1	1
18	219.4	31.04	1	1
19	204.7	28.96	1	1
20	179.9	25.45	1	1

**Failure Mode: 1 – Adhesive; 2 - Cohesive, with composite; 3 - Cohesive, with acetal resin; 4 - Mixed, with composite; 5 - Mixed, with resin. 6 - Mixed, with composite and resin.*

*** Simplified Failure Mode: 1 – Adhesive; 2 – Mixed; 3 – Cohesive.*

Data from experimental group: POM + Futurabond® M^{+}

N	Force (N)	MPa	Failure Mode	Simplified Failure mode
1	176	24.9	1	1
2	232.9	32.95	1	1
3	185	26.17	1	1
4	174.7	24.71	1	1
5	242.4	34.29	1	1
6	189.6	26.82	1	1
7	165.8	23.46	1	1
8	223.8	31.68	1	1
9	219.2	31.01	5	2
10	183.6	25.97	1	1
11	164.7	23.3	1	1
12	125.1	17.7	1	1
13	151.8	21.36	5	2
14	172.7	24.43	1	1
15	158.9	22.48	1	1
16	199.6	28.24	1	1
17	177.4	25.1	1	1
18	215.6	30.5	1	1
19	156	22.07	1	1
20	193.6	27.39	1	1

**Failure Mode: 1 – Adhesive; 2 - Cohesive, with composite; 3 - Cohesive, with acetal resin; 4 - Mixed, with composite; 5 - Mixed, with resin. 6 - Mixed, with composite and resin.*

*** Simplified Failure Mode: 1 – Adhesive; 2 – Mixed; 3 – Cohesive.*

Data from experimental group: POM + ScotchBond™ U

N	Force (N)	MPa	Failure Mode	Simplified Failure mode
1	188.8	26.71	1	1
2	169.8	23.91	1	1
3	195.5	27.66	1	1
4	208.7	29.52	1	1
5	153.9	21.77	1	1
6	169	23.91	1	1
7	107.9	15.26	1	1
8	161.8	22.89	1	1
9	189.9	26.87	1	1
10	111.6	15.79	1	1
11	179.6	25.41	1	1
12	157.1	22.23	1	1
13	162.6	23	5	2
14	134.5	19.03	1	1
15	140.6	19.89	1	1
16	175.3	24.8	1	1
17	201.2	28.46	1	1
18	178.9	25.31	4	2
19	135.9	19.23	1	1
20	174.9	24.74	1	1

**Failure Mode: 1 – Adhesive; 2 - Cohesive, with composite; 3 - Cohesive, with acetal resin; 4 - Mixed, with composite; 5 - Mixed, with resin. 6 - Mixed, with composite and resin.*

*** Simplified Failure Mode: 1 – Adhesive; 2 – Mixed; 3 – Cohesive.*

Data from experimental group: POM + OptiBond® Solo Plus™

N	Force (N)	MPa	Failure Mode	Simplified Failure mode
1	157.6	22.3	1	1
2	146.8	20.77	1	1
3	162.1	22.93	1	1
4	177.1	25.05	1	1
5	137.9	19.51	1	1
6	129.4	18.31	1	1
7	135.4	19.16	1	1
8	181.6	25.69	5	2
9	180.5	25.54	1	1
10	150.3	21.26	1	1
11	142.4	20.15	1	1
12	192.1	27.18	4	2
13	181.3	25.69	1	1
14	140.4	18.86	1	1
15	147.1	20.81	1	1
16	192.8	27.28	4	2
17	162.7	23.02	1	1
18	151.1	21.38	1	1
19	168.2	23.8	1	1
20	171.3	24.23	4	2

**Failure Mode: 1 – Adhesive; 2 - Cohesive, with composite; 3 - Cohesive, with acetal resin; 4 - Mixed, with composite; 5 - Mixed, with resin. 6 - Mixed, with composite and resin.*

*** Simplified Failure Mode: 1 – Adhesive; 2 – Mixed; 3 – Cohesive.*

Data from experimental group: PMMA + OptiBond™ XTR

N	Force (N)	MPa	Failure Mode	Simplified Failure mode
1	221.4	31.32	4	2
2	188.9	26.72	4	2
3	183.1	25.9	4	2
4	220.9	31.25	4	2
5	191.4	27.08	6	2
6	217.3	30.74	4	2
7	207.2	29.31	6	2
8	218.6	30.93	4	2
9	223.1	31.56	6	2
10	245.3	34.7	4	2
11	215.8	30.53	4	2
12	221.4	31.32	1	1
13	188.1	26.61	4	2
14	277.9	39.31	4	2
15	254.2	35.96	4	2
16	215.7	30.52	6	2
17	209.8	29.68	4	2
18	233.3	33.01	4	2
19	248.1	35.1	4	2
20	190.7	26.98	4	2

**Failure Mode: 1 – Adhesive; 2 - Cohesive, with composite; 3 - Cohesive, with acetal resin; 4 - Mixed, with composite; 5 - Mixed, with resin. 6 - Mixed, with composite and resin.*

*** Simplified Failure Mode: 1 – Adhesive; 2 – Mixed; 3 – Cohesive.*

Data from experimental group: PMMA + Futurabond® M^{+}

N	Force (N)	MPa	Failure Mode	Simplified Failure mode
1	169	23.91	4	2
2	244.3	34.56	4	2
3	270.6	38.28	4	2
4	141.3	19.99	1	1
5	230.4	32.59	4	2
6	190.5	26.95	4	2
7	249.1	32.24	4	2
8	206.6	29.23	4	2
9	185.2	26.2	4	2
10	179.6	25.41	4	2
11	239.3	33.85	4	2
12	237.6	33.61	4	2
13	243.2	34.41	1	1
14	241.1	34.11	4	2
15	187.4	26.51	4	2
16	214.5	30.35	6	2
17	187.8	26.57	4	2
18	255.9	36.2	6	2
19	203.6	28.8	4	2
20	221.1	31.28	4	2

**Failure Mode: 1 – Adhesive; 2 - Cohesive, with composite; 3 - Cohesive, with acetal resin; 4 - Mixed, with composite; 5 - Mixed, with resin. 6 - Mixed, with composite and resin.*

*** Simplified Failure Mode: 1 – Adhesive; 2 – Mixed; 3 – Cohesive.*

Data from experimental group: PMMA + Scotchbond™ U

N	Force (N)	MPa	Failure Mode	Simplified Failure mode
1	189.4	26.79	4	2
2	205.5	29.07	4	2
3	222.9	31.35	4	2
4	199.4	28.21	4	2
5	189.2	27.77	1	1
6	185.6	26.26	4	2
7	220.7	31.22	4	2
8	277.1	39.2	4	2
9	192.5	27.23	1	1
10	222.1	31.42	4	2
11	270.1	38.21	4	2
12	214.4	30.33	4	2
13	200.1	28.31	4	2
14	202.8	28.69	4	2
15	192.8	27.28	4	2
16	235.4	33.38	4	2
17	235.1	33.26	4	2
18	186.3	26.36	4	2
19	245.1	34.67	4	2
20	243.5	34.45	4	2

Failure Mode: 1 – Adhesive; 2 - Cohesive, with composite; 3 - Cohesive, with acetal resin; 4 - Mixed, with composite; 5 - Mixed, with resin. 6 - Mixed, with composite and resin.

*** Simplified Failure Mode: 1 – Adhesive; 2 – Mixed; 3 – Cohesive.*

Data from experimental group: PMMA + OptiBond® Solo Plus™

N	Force (N)	MPa	Failure Mode	Simplified Failure mode
1	198.6	28.1	4	2
2	236	33.39	4	2
3	208.1	29.44	4	2
4	170.4	24.11	1	1
5	219.9	31.11	4	2
6	217.5	30.77	1	1
7	234.4	33.16	1	1
8	203.3	28.76	4	2
9	162.3	22.96	1	1
10	163.5	23.13	4	2
11	245.3	34.7	4	2
12	236.2	33.42	4	2
13	214	30.27	4	2
14	187.4	26.51	4	2
15	232.5	32.89	4	2
16	228.4	32.31	4	2
17	212.3	30.03	4	2
18	211.1	29.86	4	2
19	195.5	27.66	1	1
20	176.1	24.91	1	1

Failure Mode: 1 – Adhesive; 2 - Cohesive, with composite; 3 - Cohesive, with acetal resin; 4 - Mixed, with composite; 5 - Mixed, with resin. 6 - Mixed, with composite and resin.

*** Simplified Failure Mode: 1 – Adhesive; 2 – Mixed; 3 – Cohesive.*

Instructions for use (Grandio® SO)

VOCO

USA

Grandio®SO

Universal nano-hybrid restorative

in accordance DIN EN ISO 4049

CE 0482

Carefully read instructions prior to use

GrandioSO is a light-curing, radiopaque restorative. **GrandioSO** contains 89% w/w inorganic fillers in a methacrylate matrix. **GrandioSO** is used with a dentineenamel bond and is available in syringes and caps for direct application.

Shades:
A1, A2, A3, A3.5, A4, B1, B2, B3, C2, D3, OA1, OA2, OA3.5, BL, GA3.25, GA5

Handling:
Allow the material to reach room temperature before use.

Bond material:
GrandioSO is used in the adhesive technique with a dentine-enamel bond. All light-curing bond materials can be used. Follow the respective instructions for use with regard to preparation (etching technique) and application.

Application of GrandioSO:
Apply the selected shade of **GrandioSO** in layers that are a maximum of 2 mm thick, adapt with a suitable instrument and light-cure afterwards.

Light-curing:
Conventional polymerization devices are suitable for light-curing the material. The curing time is as follows when using an LED-/halogen light with a minimum light-output of 500 mW/cm²

20 s for shades A1, A2, A3, A3.5, A4, B1, B2, B3, C2, D3, BL, GA 3.25
40 s for shades OA1, OA2, OA3.5, GA5

The curing time is as follows when using an LED-/halogen light with a minimum light-output of 800 mW/cm²

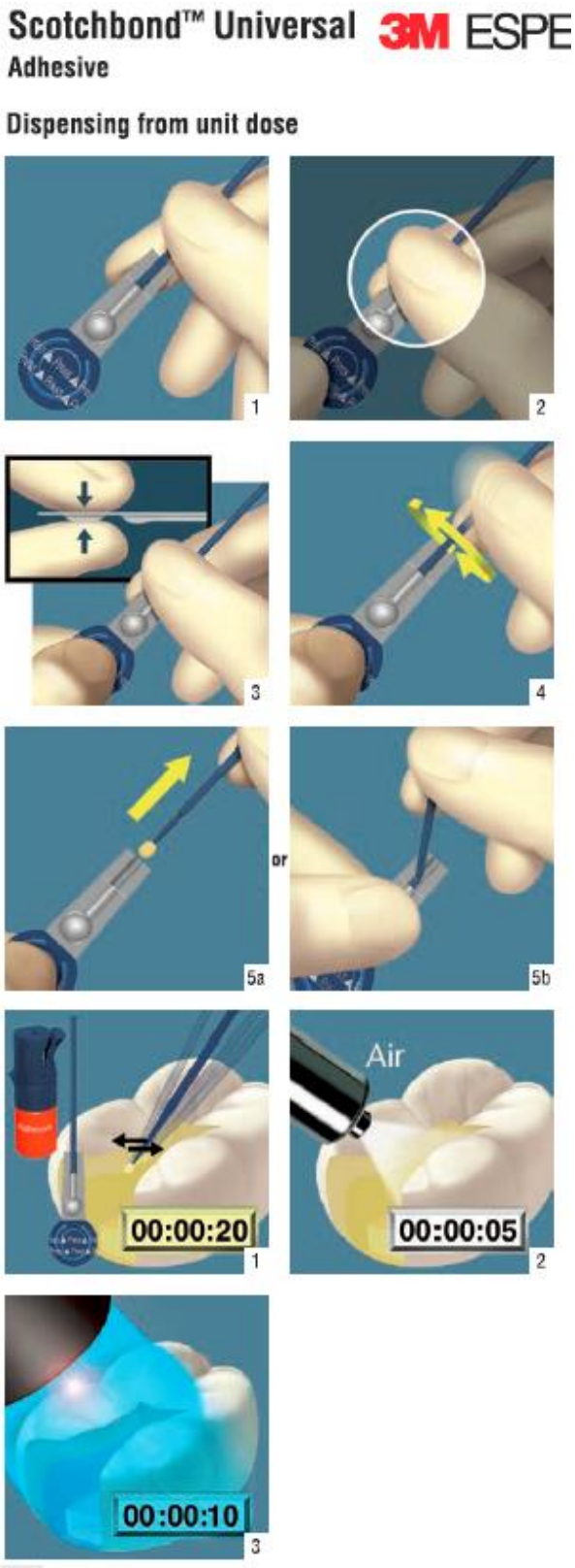
10 s for shades A1, A2, A3, B1, BL
20 s for shades A3.5, A4, B2, B3, C2, D3, GA3.25, GA5
40 s for shades OA1, OA2, OA3.5

Hold the light emission tip of the device as close as possible to the surface of the filling. Otherwise, the curing depth may be reduced. Incomplete curing may lead to discoloration and discomfort.

Instructions for use (OptiBond™ XTR)

Instructions for use (Futurabond® M^{+})

Instructions for use (Scotchbond™ U)



Instructions for use (OptiBond® Solo Plus™)

OptiBond®
Solo Plus™
Kerr
Sybron Dental Specialties

Bottle delivery:

- Shake bottle briefly prior to dispensing.
- Dispense one drop of adhesive into disposable mixing well.
- Wet applicator tip completely.

1. Apply OptiBond Solo Plus with light brushing motion for 15 seconds.
2. Air thin for 3 seconds.
3. Light cure 20 seconds.